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#### Functional properties of food and non-food soy protein-based products

by

Ee-Fah Chong

A thesis submitted to the graduate faculty in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE

Major: Food Science and Technology Major Professors: Jay-Lin Jane and Lester A. Wilson

Iowa State University

Ames, Iowa

2000

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This is to certify that the Master's thesis of

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has met the thesis requirements of Iowa State University

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#### ABSTRACT

The major odorants from soy protein-based pellets and foams were investigated. Soy-based pellets were extruded from commercial soy protein isolates at 100°C. The pellets were thermally extruded at 150-160° to produce soy-based foams. A Tenax adsorbent was used to trap and concentrate volatiles. Gas chromatography-mass spectrocopy identified volatile compounds including aldehydes, alcohols, ketones, and aromatic hydrocarbons in the pellets and foams. Pyrazines were found only in the soy-based foams. Compounds with significant odors were identified and their odor intensities were evaluated by using gas chromatograohy-olfactometry. Toluene and hexanal were found to be the strongest odors in the pellets and foams. The intensities of odoriferous compounds in the foams were greater than those from the pellets. Lipoxygenase-free soy protein isolates and masking agents could reduce undesirable odor of the soy-based plastics.

The effects of storage temperature and food pH's on foaming, oil absorption and water-holding capacity of full-fat soy flakes were also investigated. Foaming properties were measured by whipping protein suspensions. Oil absorption was measured by mixing, centrifuging and determining the oil adsorbed by soy flakes. The measurement of water-holding capacity involved the application of centrifugation to separate retained water from free water. Foam expansion of the flakes was enhanced by high storage temperatures whereas foam stability decreased with high storage temperatures. Foams have the lowest stability at pH 3.5 and 9.0 and the highest stability at pH 5.5. Oil absorption of the flakes was the highest at 50°C after 7 days of storage. The flakes stored at 4°C and 25°C had still not reach the same level of oil absorption in 28 days as had the flakes stored at higher

v

temperatures. High water-holding of the flakes was minimum at pH 6.5 and maximum at pH 9.0. There was no significant difference for the functional properties of the soy flakes stored at 4°C for 28 days. High storage temperatures, i.e. 45 and 50°C, have more pronounced effects on the quality of soy flakes. The results indicated that transit times during shipment of up to four weeks and storage temperature of soy flakes below 25°C could provide optimum quality in the flakes.

#### **GENERAL INTRODUCTION**

#### Introduction

Soy proteins have a wide range of utilization in both non-food and food industries. In non-food usage, soy proteins can produce household products such as foamed clamshell container or trays, beverage cups, utensils, trash bags, or thermal insulation products at a competitive price. The products are environmental friendly and are derived from renewable resources.

One of the non-food usages of soy protein is to produce soy-based polymeric packaging material. Biodegradable plastics are an alternative approach to address the solid waste and marine pollution problems. The plastics can replace non-biodegradable polymers such as polyethylene and polystyrene. In addition, replacing petroleum-based synthetic plastics with biodegradable plastics can decrease the release of carbon dioxide.

However, the undesirable odor of the soy-based plastics limits their acceptance by consumers. The compounds responsible for the undesirable odor characteristics of soy protein products are mainly aldehydes, furans, alcohols, ketones, and phenolics (1-2). The most commonly encountered undesirable odors from soy products have been identified as beany, grassy, and earthy (1-5). At present, there is little information on the analysis of volatile compounds of soy-based plastics. Thus, the first part of the study was to investigate major odorants in soy-based pellets and foams.

For food applications, soy proteins such as soy flakes, soy concentrates or soy isolates are used to produce meat analogs, coffee whiteners, beverage products, breakfast cereals or bakery products. They have been used to supplement or replace ingredients derived from animal sources or used to improve the quality of food systems.

Functional properties such as foaming, water-holding capacity and oil absorption capacity play an important role in incorporating soy proteins into food systems (6-8). The properties can impart desirable or undesirable attributes such as appearance, texture, viscosity, and mouth feel in various food products. Functional properties of soy protein products have been studied extensively (6-10). However, studies of the changes on functional properties of full-fat soy flakes during storage are limited. The second part of the study was to investigate the influence of temperature and food pH's on functional properties of full-fat soy flakes after different storage periods.

#### **Thesis Organization**

This thesis consists of a general introduction, a literature review, two manuscripts and a general conclusion. The first paper is entitled "Major Odorants in Soy Protein-Based Pellets and Foams." The second paper is entitled " The Effects of Storage Temperature and End Use pH's on the Functional Properties of Full-fat Soy Flakes." The papers will be submitted to the Journal of the American Oil Chemists' Society.

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#### LITERATURE REVIEW

#### Soy Proteins

Soybeans contain about 44 to 50% protein. Soy proteins found in soy flakes, soy flour, soy protein concentrates and soy protein isolates, can be used as food ingredient or processed directly into soybean products. In addition to proteins, lipids (20%), carbohydrates (30%), moisture (4-10%), minerals, phosphorus and minor compounds such as phenolic compounds, isoflavones and saponins are also found in soybeans (1). The main carbohydrate constituents in soybean included glucose, stachyose, raffinose, sucrose, hemicellulose and cellulose. The basic steps in processing soybeans for human and pet foods are cleaning, cracking, dehulling, tempering, flaking and extracting (2).

Soy flakes are produced by dehulling the soybeans. The dehulled soybeans are dried and flaked to produce 50% protein full-fat soy flakes. The full-fat flakes can be hexane extracted to produce soybean oil and defatted white flakes. The white flakes are then further processed to produce a variety of protein products for animal and human foods. According to the Soy Protein Council (2), defatted soy flakes can be divided into different categories based on nitrogen solubility index (NSI) such as white flakes (>85 NSI), cooked flakes (20-60 NSI), and toasted flakes (<20 NSI).

Soy flours (<100 mesh) and soy grits (>100mesh) are prepared by first removing the hulls from the cleaned beans. The beans are then dried, tempered, ground, and sifted to produce full-fat soy flour or soy grits (2, 3). To produce defatted soy flour or soy grits, the dehulled beans are conditioned, flaked, solvent extracted, desolventized and dried. Defatted soy flour contains about 40-50% protein, 38% total carbohydrates, and 6% ash (4).

Soy protein concentrates contain about 70% protein (2). Soy concentrates are prepared from defatted flakes or flour by removing carbohydrates, ash, peptides and phytic acids (5). 20-80% aqueous alcohol extraction and dilute aqueous acid leaching at pH 4.5 are two of the most commonly used processes to produce soy concentrates. The nitrogen solubility index of soy concentrates in the acid leaching process (65-75% NSI) is relatively high because severe denaturation steps are not introduced in the process (2). Wang and Murphy (6) reported that alcohol washed commercial soy concentrates contain low concentration of isoflavones. Alcohol washing removed most of the isoflavones along with the soluble carbohydrates such as raffinose and stachyose that responsible for flatulence.

Soy protein isolates contain more than 90% protein. They are produced from the defatted soy flakes or flour, which are extracted with dilute alkali at pH 7-9 (7). Then, alkaline insoluble protein and carbohydrates are removed by using filtration or centrifugation. The clarified extract (i.e. protein, sugar, ash and minor components) is acidified to pH 4.5, which is the isoelectric point of most of the soy proteins. It causes separations of supernatant (sugar, ash, etc.) and precipitated proteins (7). The proteins are washed, neutralized and spray dried. Soy protein isolates are widely used for their emulsifying property, viscosity, foaming capacity and adhesion property.

The physical and chemical properties of soy proteins are affected by soybean variety, growing environment, type of processing, and storage conditions. Baker et al. (8) demonstrated that 70% ethanol extracted defatted soy flours at 30°C have significantly lower undesirable odor than the full-fat soy flakes. Traina and Breene (9) showed that the foam stability of enzyme active soy flours was higher than that of the enzyme-inactive soy flours. Rakosky (5) reported that soy protein extraction could cause protein denaturation.

#### Usage of Soy Proteins: Non-food and Food Usage

According to Myers (10), only 0.5 % of soy proteins are used for non-food purposes. Soy proteins are inexpensive and are desirable to produce disposable products such as trash bag, utensils, food containers, mulch and agricultural films. In the 40's, Henry Ford promoted the usages of soy proteins in industrial products (10). Soy-based household appliances, automobiles, and tools were produced under Henry Ford's guidance.

Over 21.9 billion pounds of plastics were disposed of in 1992 (11). The majority of the plastics was polyethlylene and polystyrene-based. The synthetic plastics are used due to their safe, strong, light-weight and economical features. However, the plastics cannot degrade in the environment. Ecological and environmental concerns kept researchers interested in applying soy protein as a biopolymer source. Once soy protein-based polymers are degraded, the residuals can be recycled or reused as soil fertilizers or animal feeds.

In the food area, soy proteins are used in diary analogs, textured soy proteins, meat analogs, fermented and nonfermented soy foods. Until 1992, only about 3% of soy proteins were used for human foods because of their undesirable flavor and the presence of flatulence factors (12). Now, soy proteins are gradually being accepted in the food industry because they provide desirable functional properties in foods at a lower cost than animal source alternatives such as dried milk solid. In addition, soy foods are nutritious (2, 12). The Food and Drug Administration in 1999 authorized the use of health claims on food labels about the role of soy proteins in reducing the risk of coronary heart disease. Foods containing soy protein in a low fat, low saturated fat and cholesterol diet could lower blood cholesterol levels (13). However, a food must contain at least 6.25 g of soy protein per serving and less than 3 g of fat in order to qualify for the health claim. The applications of soy products in

foods included doughnut mixes, breakfast cereals, bakery products, baby foods, soups, sauces and gravies, and comminuted meat products.

#### **Functional Properties of Soy Proteins**

Functional properties of soy proteins are crucial in food applications. These properties reflect the composition and conformational change of protein molecules and the interactions between proteins and fats, carbohydrates, and other proteins (4, 14). The properties can be affected by food formulation, food processing, packaging, storage and shipping conditions. These functional properties rely on the changes in temperature, pH, organic solvent and protein concentration.

Functional properties, such as foaming properties, oil adsorption capacity and water holding capacity, are important factors in delivering desirable texture, flavor, and stability to food products in order to be accepted by consumers. Table 1 shows the functional properties of soy proteins (4). In foaming properties, a foam can be described as two-phase systems where the gas bubbles phase is surrounded by a continuous liquid phase (15). Soy protein can form foams. During foaming, the protein concentrates at the gas bubble and liquid interphase where it reduces the surface tension and resists migration back into the water phase. Foam expansion describes the ability of protein to develop foams (4, 15). The property is associated with the amount of interfacial area on protein molecules. Foam stability refers to the ability of protein molecules to stabilize foam against gravitational and mechanical stresses. Foaming properties are critical in producing icing, whipped toppings, frozen desserts and various types of cakes.

Functional property	Mode of action	Food system	
Foaming	Forms stable films to entrap gas	Desserts, angel cakes, whipped toppings	
Oil adsorption	Binding of free fat; entrapment of oil	Sausages, meats, doughnuts, simulated meats	
Water holding	Hydrogen-bonding of H <sub>2</sub> O; entrapment of H <sub>2</sub> O	Sausages, meats, simulated meats, breads, cakes	

Table 1. Functional properties of soy proteins in food systems

The main experimental approaches in measuring foaming properties included bubbling, whipping/beating and shaking. The basic foam formation is the creation of gas bubbles in liquid. In the bubbling method, gas bubbles are forced through a solution and a column of foam is allowed to form above the solution (16). The method is reproducible and gives uniform bubble size.

Whipping can be performed through gas introduction by vigorously agitating a liquid. The whipped foam is well mixed throughout its formation. Since the volume of air included increases with the intensity of beating, the maximum levels of gas incorporation during whipping reflects a more real dynamic equilibrium between mechanical formation and destruction of bubbles (15-16). Whipping is a practical production of foams. In the whipping method, agitation is continued for a fixed amount of time and proteins tends to produce greater number of bubbles (16).

The shaking method is rarely used in foam measurement. The rate of gas introduction into a solution depends on the frequency and the amplitude of shaking, the shape of the container, the volume and the flow properties of the liquid (15). Foam formation by shaking tends to be slower than the bubbling and whipping methods. There is no systematic study on form formation.

Eldridge et al. (17) demonstrated that minimum foam stability and foam expansion of alcohol-extracted soy isolates were found between pH 4 to pH 6. Horiuchi et al. (18) reported that heating soy proteins at 70-80°C could enhance foaming properties. The presence of lipid compounds in soy products could destabilize the protein films which can be detrimental to the foaming properties in soy proteins (19).

Oil absorption is considered to be a physical oil entrapment during the addition of excess oil to protein products (4). The measurement of oil absorption includes thorough mixing, centrifuging and determining the oil adsorbed by soy proteins by weighing. The amount of absorbed oil is measured by the total amount of oil minus the free oil (4, 20). The type of oil, the amount of oil and protein in the sample, and the centrifuging condition would affect the oil absorption of proteins (20, 21). Voutsinas and Nakai (22) developed a turbidimetric method for determining the fat binding capacity of proteins. The turbidity was dependent on wavelength, blending time, and volume of oil.

Torgersen and Toledo (23) found that the more soluble the protein the less the fat binding capacity in comminuted meat system. One of the reasons suggested by Voutsinas and Nakai (22) is soluble proteins might allow less hydrophobic binding sites to be sterically available for interaction with oil hydrocarbon chains. The addition of soy proteins in doughnuts decreases oil absorption (24, 25). According to Lin et al. (20), fat adsorption of soy proteins is little affected by pH or temperature but is closely related to protein content. Oil absorption is important in producing simulated meat, dairy analogs, and bakery products.

Water-holding capacity denotes the maximum amount of water that can be absorbed or retained by protein materials under various food conditions (4). It indicates the interaction of polar side chains of soy proteins with water molecules in the food system. Water absorption capacity is an important property in making simulated meats, breads and cakes.

Water-holding capacity can be determined by two popular techniques. The first one involved the application of centrifugation to separate the retained water from the free water (26, 27). The supernatant is decanted and the absorbed water is measured by either weight differences or volume differences. The other way of estimating water absorption of protein was developed by Hermansson (28). A protein sample is dusted on a wetted filter paper fastened on a glass filter. It is placed on the top of a funnel filled with water and the devise is connected to a circular capillary. The amount of water absorbed by the sample can be measured by observing the capillary. In this technique, the thickness of sample on the wetted filter paper is critical to obtain reproducible results. Both methods involved some losses of soluble proteins.

Fiora et al. (29) demonstrated that the type of processing and the composition of protein affected the water holding property of soy protein isolates. Johnson (30) showed that heat treatment enhanced water-holding capacity of soy flours.

#### **Odor in Soy Proteins**

Soybean products contain undesirable odors that limit their acceptability in non-food and food markets especially when used in large amounts. Volatile compounds in soybeans and soy products are mainly responsible for characteristic odors. Some of these odors are acceptable, however, the majorities of the odors are undesirable and offensive. Apart from

the inherent odors such as grassy, green and beany characteristics from soybean and soy products, different odors can be produced from processing and storage conditions (31).

The chemical classes that contribute to odorous volatile compounds in soybeans and soy products are aldehydes, ketones, alcohols, furans, phenolics and amides (31-32). Volatile compounds associated with odor characteristics in soybeans, soybean oil and soy products are shown in Table 2 (31).

Odor description	Volatile compounds
Green, grassy, beany	Pentanal
	Hexanal
	Decanal
	2-Hexenal
	2-Pentylfuran
	Pentanol
	Hexanol
	1-Octen-3-ol
	3-Octen-2-one
Roasted	Alkylpyrazines
	4-Ethyl-2-methylthiazole
	Maillard reaction products
Oily/fatty, tallow-like	Hexanal
	Octanal
	Nonanal
	3-Octen-2-one
	Non-2-enal
Musty, moldy, earthy	1-Octen-3-ol
	Acetophenone
Mushroom	1-Octen-3-ol
	3-Octen-2-one
Oxidized, cardboard-like, oily, paint-like	Higher alka-2, 4-dienals (e.g. C7, C8, C9, C10)

Table 2. Odor description of volatile compounds in soybeans, soybean oil and soy products

Doi et al. (33) identified 50 compounds from roasted soybeans. Hexanal and 1hexanol, which imparted beany flavor, decreased with roasting. However, pyrazines, furans and pyrrole increased with roasting. Boatright and Crum (34) studied the odorous volatile compounds in commercial soy protein isolates. Hexanal, butyric acid, 2-pentyl pyridine, and 2-methyl butyric acid methyl ester were described as having oxidized and nutty, sweaty feet, penetrating and grassy, and fruity, respectively. The compounds were considered the major odorous compounds in the isolates.

Kobayashi et al. (35) studied the aroma constituents of soybean milk lacking lipoxygenase isozymes (LOX) by using simultaneous distillation and extraction (SDE). The amount of volatile compounds decreased with the removal of lipoxygenases. However, the amount of 1-octen-3-ol found in three types of soybeans (i.e. LOX-1, 2, 3; LOX-2, 3-null; LOX-1, 2, 3-null [free]) was similar to each other. In addition to lipid oxidation, this compound is also formed by enzymatic reaction (i.e. hydroperoxide lyase) which is presented in these types of soybean cultivers (35). Soybeans lacking lipoxygenase isozymes produce limited amount of aldehydes and alcohols (36). Qvist and van Sydow (37) found that the concentrations of aldehydes and sulfur compounds in heated soy protein model system increased with heat.

The primary source of undesirable odors in soybean and soybean products are mainly due to lipid oxidation. Maillard reactions and Strecker degradation of amino acids can impart both desirable and undesirable odors (31). The Maillard reactions are induced by heat. It is the interactions of a free amino group with carbonyl compounds (31, 38). The Maillard reaction enhanced as reaction time and temperature increased. The resulting compounds formed by the Maillard reactions included aldehydes, furans, ketones, alcohols,

pyrroles, pyridines, and pyrazines (31, 38). Strecker degradation involves the oxidative deamination and decarboxylation of an amino acid in the present of a dicarbonyl compound (31, 38). The compounds produced from the degradation reaction included acetaldehyde, propanal, and butanal. Enzyme-catalyzed oxidation and lipid oxidation with precursors such as linoleic and linolenic acids could produce hydroperoxides. These compounds will form undesirable products including hexanal and 2-hexenal (31).

#### Gas Chromatography Headspace Analysis

Gas chromatography analysis is broadly used in the detection of amino acids, fatty acids, vitamins, pesticides, food additives, flavor and odor compounds. The analysis provides qualitative and quantitative information about the volatile compounds. Headspace methods can be divided into direct injection and dynamic headspace concentration (39-41). Direct injection is one of the simplest methods of isolating volatile compounds. It is a direct injection of vapors or gaseous mixture from a sample within a closed system (39). It is rapid and it requires minimal sample preparation. In addition, the results from the direct headspace analysis can be related to the perception of compounds by the human nose.

Cryogenic trapping is part of the direct injection method. In cryogenic focusing, a cryogenic coolant (e.g. liquid nitrogen or dry ice/acetone) is used to focus volatiles in the column (42). It increases the capacity factor and improves peak shape. The technique slows down compound migration through a column (40). A cryogenic trap will collect volatiles despite the polarities and boiling points of the compounds. Only high concentrations of volatiles might be detected by direct injection (40, 42). Thus, one must collect an adequate of sample to produce a detectable response.

Some of the problems of using direct injection are the condensation of volatiles inside the sampling syringe, and the absorption of volatiles in the septum of the sampling container (41). Food samples mainly contain moisture. The method detects the most abundant volatile compounds. In addition, a large volume of sample is needed to produce a detectable response due to the low concentration of volatiles in a sample.

The dynamic headspace concentration involves passing headspace vapors either through a cryogenic trap or adsorption trap. In the analysis, a sample is placed in a volatile glass trap. The volatile compounds from the sample are purged with an inert gas. The aforementioned process is to obtain a large volume of volatile compounds for the headspace analysis (43). The cryogenic trapping in the dynamic headspace is similar to that of direct injection method.

In adsorption trap method, an absorbent is used to trap and concentrate volatile compounds. The trapping material usually has little affinity for water. The adsorbent such as charcoal, Porapak Q or Tenax is used to concentrate volatile compounds (39). Charcoal (coconut carbon) strongly adsorbs nonpolar compounds but weakly adsorbs polar compounds such as water molecules. The surface area of the absorbent is 1150-1250 m<sup>2</sup>g (44). It has a very large adsorption capacity if compare to other synthetic polymers. For example, Porapak Q that is made from ethyl vinyl benzene-divinyl benzene, retained less ethanol than charcoal (44). Porapak Q has a surface area of 550-650 m<sup>2</sup>g. The disadvantage of using charcoal as an absorbent is the impure carbon could produce artifacts during the thermal desorption of the analysis (41, 43).

Tenax absorbent is made from diphenyl-phenylene oxide. The surface area of Tenax is  $18.6 \text{ m}^2\text{g}$  (44). Tenax absorbent is one of the most commonly used synthetic porous

polymers in headspace analysis. The adsorbent showed good thermal stability and adsorption capacity. It also exhibits good recovery of absorbed volatiles. The adsorbed volatiles can either be solvent extracted or thermally desorbed with the assistance of the purged inert gas (e.g. helium). However, the disadvantage of using adsorbent trap is its absorption affinity (40, 42). According to Sydor and Pietryzk (44), charcoal can absorb 24.7% capacity (percentage of weight of absorbent) of benzene while Tenax polymer only can trap 0.53% capacity of benzene. In the dynamic headspace analysis, sampling time, purge gas flow rate and trap capacity could affect the results of chromatograms representing the sample.

In the identification of volatile compounds by gas chromatography analysis, numerous detectors are available for the method, including thermal conductivity detector (TCD), flame ionization detector (FID), electron capture detector (ECD), flame photometric detector (FPD) and nitrogen-phosphorus detector (NPD). The TCD can detect almost anything including water molecules. However, it has poor sensitivity. It is not broadly used in food applications except the lack of response of other detectors towards the water molecules, CO or CO<sub>2</sub> (39). In addition, specificity in detector response for certain compounds is often preferable in GC analysis (39, 43).

The FID can detect most of the organic compounds. It has good sensitivity in organic compounds (39). The FID is widely used in food applications, such as flavor studies, carbohydrate analysis and fatty acid analysis.

The specificity of ECD is to detect halogen, nitrogen, phosphorous, sulfur, metals or conjugated double bonds compounds. The detector is widely used in pesticide residue determinations (39-40). Some selective detectors such as FPD and NPD are specialized in

sulfur/phosphorous and phosphorous/nitrogen compounds detection, respectively. The FPD and NPD are widely used in pesticides analysis and flavor studies.

GC equipped with detectors can provide the amount and the retention time of a volatile compound. Identification of a compound is needed by comparing its response and retention time with its standard. The compound identification can be supplied by mass spectrometer (MS). The MS analysis provides the molecular weight and molecular structure of a compound. The GC/MS is one of the most popular methods for flavor analysis.

#### Sensory Evaluation

According to Lawless and Heyman (45), sensory evaluation is defined as a method to measure, analyze and interpret the true evaluation of the sensory impact (i.e. sight, flavor, touch, or hearing) of a product. It evaluates the quality of a product by avoiding bias effects and collecting crucial perceptions from the potential consumers. Sensory evaluation can be divided into four categories: discriminative tests, descriptive tests, attribute difference tests and affective tests (45-46). Discriminative tests are used to determine product differences including triangle test, duo-trio test, and sequential test. Descriptive tests are designed to measure product differences in specific sensory characteristics (e.g. texture, odor, taste, and color). Some of the descriptive tests included flavor profile, texture profile, and time-intensity descriptive analysis.

Attribute difference tests are used to determine the existence of a difference or the degree of differences in two or more samples with respect to a defined attribute. The attribute can be saltiness, freshness, or lightness (45-46). Some of the commonly used

attribute difference tests are paired comparison test, simple ranking test and pairwise ranking test.

Affective tests can be divided into acceptance tests and preference tests. In acceptance tests, the products are rated on the basis of acceptability. While, in preference tests, the products are tested in the order of preference. Affective tests are used to measure the acceptance and preference of a product by consumers (47).

Odor analysis is considered as one of the descriptive tests. The panelists should be well trained to produce consistence results. In addition, the panelists should be able to describe the characteristics and intensities of odor compounds.

Gas chromatography olfactometry (GCO) is widely used in odor analysis. It is to determine the potency of odorants in food extracts. GCO technologies can be divided into four categories: sniffing GC effluent after separation of individual volatile compounds, aroma extract dilution analysis (AEDA), CharmAnalysis<sup>TM</sup>, and Osme. In the GCO analysis, the volatile compounds are sniffed by human nose from the detector port of GC. A descriptive response including characteristics and intensities of the odor compounds is correlated to the peaks of a chromatogram (48).

The quantitative analysis of GCO can be achieved by the aroma extract dilution analysis (AEDA). It is a series of diluted flavor extracts and it provides a flavor dilution factor. The dilution at which a volatile compound can still be detected is defined as its flavor dilution (FD) factor. The higher the potency of a volatile compound, the higher the dilution factor in an extract. The FD factor is a relative measurement. Kobayashi et al. (35) used AEDA to measure odors in soybean milk. The yields of volatiles in soy milk such as hexanal and 2-pentylfuran was greatly reduced when the lipoxygenases in soybean were lacking.

Charm Analysis<sup>TM</sup> determines odor compounds in a mixture by sniffing the GC effluent through a series of dilutions (49). Charm Analysis<sup>TM</sup> yields chromatograms in which the ordinates associate with odor potency or odor threshold. The plot of Charm Analysis<sup>TM</sup> is the dilution value (*v*-axis) versus retention index (*x*-axis). Sensory descriptions of the odor also are recorded. The dilution value is analogous to the flavor dilution in AEDA plot. Moio et al. (50) studied the odor impact of raw, pasteurized and UHT bovine milk by using Charm Analysis<sup>TM</sup>. Dimethyl sulphone and indole were found to have odor impact in all types of milk.

Osme is a quantitative method used to measure the perceived odor intensity of a volatile compound eluting from a GC olfactometer (51). It is a time-intensity approach evaluation. An odor peak is provided and verbal descriptions of the peak are recorded. The method of analysis for Osme is not based on odor detection thresholds such as AEDA and CharmAnalysis<sup>TM</sup>. The Osme method has been used to study the odor analysis of wines (51-52). Miranda-Lopez et al. (52) studied the odor analysis of wines from grapes of different maturities by Osme. The authors demonstrated that wines harvested at the end of ripening period had more odors than wines from earlier harvested fruit.

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## MAJOR ODORANTS IN SOY PROTEIN-BASED PELLETS AND FOAMS

A paper to be submitted to the Journal of the American Oil Chemists' Society Ee-Fah Chong, Earl G. Hammond, Lester A. Wilson and Jay-Lin Jane

#### Abstract

Commercial soy protein isolates were thermally extruded at 100°C to produce soy protein-based pellets. The pellets were extruded at 150-160°C to make soy protein-based foam. Dynamic headspace analysis was used to collect the volatiles in Tenax. The volatiles were analyzed by gas chromatography-mass spectrometry (GC-MS). Aldehydes, alcohols, ketones, aromatic hydrocarbons and pyrazines were identified. Pyrazines were found only in the soy protein-based foam. Gas chromatography-olfactometry revealed that the strongest odors could be attributed to toluene and hexanal. The intensities of the odors increased with extrusion temperature.

#### Introduction

With the increased awareness of environmental problems and global warming, there is increased interest in using biodegradable plastics as replacements for petroleum-based polymers. One of the practical concerns of using soy protein-based packaging materials is the presence of an undesirable odor, which could deter their acceptance by consumers. Little information is available on the analysis of volatile compounds of soy-based plastics. Synthetic polymers such as polyethylene, polystyrene, and polypropylene are widely used in the packaging. Karska et al. (1) reported that thermal degradation products of polyvinylchloride films were affected by processing temperature, but a sensory evaluation of the degradation products was not reported. Kim et al. (2) studied the volatile compounds found in polyvinylchloride films.

Dynamic headspace analysis and gas chromatography-mass spectrometry (GC-MS) have been used to provide qualitative and quantitative information about the volatile compounds associated with soy proteins and soy protein-based products (3, 4, 5). Gas chromatography-olfactometry (GCO) has been applied to the identification and intensities of volatile compounds in soybean isolates and soy milk (5, 6).

This investigation was undertaken to separate and identify volatile compounds from soy protein-based pellets and the resulting soy-based foam by gas chromatography-mass spectrometry (GC-MS). Major odorants and their intensities were noted. In addition, the effect of thermal processing on the volatiles in the intermediate product (soy protein-based pellets) and the finished product (soy protein-based foam) was evaluated.

#### **Materials and Methods**

#### Materials

Soy protein-based pellets and soy protein-based foams were prepared and provided by Jane et al. (7). The soy protein-based pellets processed from raw materials went through an extrusion process at 100°C. The foam was processed from the pellets at 150-160°C. Soy protein-based pellets and the resulting foam were made from soy protein isolate, salt, glycerol and water. Chemical standards were purchased from Aldrich (Milwaukee, WI) except for 2-pentylfuran (TCI America; Portland, OR) and decanal (Fluka; Ronkonkoma, NY).

#### Sample preparation

For the collection of volatile compounds, soy protein-based pellets were ground uniformly in a grinder model Quaker City Mill F No. 4 (Quaker City, PA) and soy proteinbased foam was cut by scissors to produce 0.6 cm<sup>2</sup> uniform size samples.

#### Collection of volatile compounds

The volatile compounds from 15-g of soy-based pellets or the resulting foams were collected in a glass volatile-stripping apparatus at 75°C with helium at 75ml/min according to the method of Lee et al. (8). The volatiles were trapped in a 3-mm o.d. x 72-mm tube filled with 43 mg of Tenax. The volatiles were collected for 40 min. After each dynamic headspace sampling, the Tenax tube was conditioned in the inlet of a GC (HP 5890 Series II; Hewlett- Packard, Palo Alto, CA) at 280°C for 3 hours before reuse.

#### Desorption of volatiles and GC-MS analysis

After the collection of volatiles from the Tenax tube, the tube was inserted in the GC inlet at 150°C and the volatiles were separated on a Supelco SPB-1 fused-silica capillary column (30 m, 0.25 mm i.d., 0.25  $\mu$ m film thickness) with helium at 1.7 ml/min. The volatiles were condensed on the first loop of the column for 2 min by using dry ice. Oven temperature was programmed from 50°C to 110°C at 5°C/min with initial and final holding times of 2 and 5 min, respectively. Peaks were detected by a flame-ionization detector

(FID) held at 200°C. Volatiles were identified by mass spectrometry (HP 5970 series Mass Selective Detector). Volatile compounds were identified by comparing their retention times and mass spectra with those of authentic standards. Tentative identifications were made by matching mass spectra of unknown with those in the NBS mass spectral library or published data. Each treatment was analyzed in triplicate.

#### Gas chromatography-olfactometry

Odor evaluations were made by four panelists. In preliminary sessions, samples of odorous compounds identified by GC-MS were presented to the panelists; verbal descriptors for them were discussed. In addition, a list of the descriptors (4-5, 9) frequently reported was presented to the panelists. Additional descriptors also were generated during the GCO analysis. The terms that were most frequently used to describe the various odors were selected.

The panelists sniffed individual volatiles from the GC detector port. The intensity of the compounds and sensory descriptors were recorded. A subjective intensity ranking from 0 = undetectable to 5 = strong was recorded for each volatile compound. The GC temperature program for odor evaluation was the same as that used for GC-MS analysis except the oven temperature was programmed at 1°C/min to allow a better separation of the volatile compounds to the panelists. The odor evaluation was done in duplicate.

#### **Results and Discussion**

#### Gas Chromatography-Mass Spectrometry (GC-MS)

A wide range of chemical classes was isolated from the soy protein-based pellets and foams (Table 1). The identified volatiles included aldehydes, alcohols, ketones, aromatic hydrocarbons, and pyrazines. The soy-based pellets and the soy-based foams generated similar products as the textured soy protein, including toluene, 2-pentylfuan, limonene, nonanal, and decanal (4). Some GC peaks were not further identified by MS. None of these had detectable odors.

The amount of aldehydes, alcohols, and ketones were greater than that of in the pellets (Fig. 1, Fig. 2) which may due to processing temperature (i.e., 150-160°C vs. 100°C). In addition, foams have a larger surface area than that of pellets even though identical sample weight was studied. The foams released the volatiles more readily than the pellets. The formation of these compounds can be attributed to the heating and extrusion process via thermal degradation of sugars, pyrolysis of amino acids, Strecker degradation, Maillard reactions and lipid oxidation (9). Ingredients used in formulation may alter the amount of off-odor volatiles.

Hexanal, nonanal, decanal and 2-pentylfuran can be formed through lipoxygenase oxidation or autoxidation of unsaturated fatty acid such as linolenic acid (6, 9, 10), and such lipid oxidation products have been identified frequently in soy proteins. Limonene was identified in soy-based pellets and soy-based foam by comparison of its mass spectra with authentic standard. Kao et al. (11) suggested that heat degradation of sterols or squelene in soybean oil could produce limonene, which has been identified in soybean oil, soy protein products, and citrus oil (4, 11, 14). 2-Pentylthiophene was identified in both soy-based samples (Table 1). This compound has been identified in canned beef, soy protein isolate and popcorn (12). Thiophenes have been reported as the products of thermally induced interaction of sulfurcontaining amino acids and carbohydrate (12). Mussinan and Katz demonstrated that thermal processing of foods can results in the formation of thiophenes (13).

2,6-Dimethylpyrazine and 3-ethyl-2, 5-dimethylpyrazine were identified only in the soy-based foam. The degradation of sugars and the pyrolysis of proteins can produce pyrazines (9). Boatright and Crum (5) suggested that nitrogen from the pyrolysis of proteins in the Maillard reaction contributed to pyrazine compounds. The composition of the thermal degradation products was affected by the processing temperature.

#### Gas Chromatography-Olfactometry (GC-0)

All compounds with significant odors were identified and their odor intensities were evaluated. Some of these compounds are known to posses strong odors. The odors of the compounds that were identified were confirmed by comparing their odors with those of authentic standards. In general, the panelists noticed the intensities of odorous compounds isolated from the soy protein-based foam were greater than those from the soy-based pellets. Compounds with significant odor characteristics were evaluated.

Aromatic compounds such as toluene and benzaldehyde were identified in both soybased pellets and the resulting foam (Table 2). Panelists described toluene as having a "chemical solvent or burned plastic" attribute. Toluene might arise from the thermal degradation of glucose or carotenoids, the pyrolysis of phenylalanine/tyrosine or the heat degradation of glucose and cysteine (9). Benzaldehyde is known for its nutty or almond aroma (14). Lipid oxidation or the heat degradation of phenylalanine/cysteine and glucose was one of the probable pathways to produce benzaldehyde (9). Toluene and benzaldehyde have been reported in polyvinylchloride films (2).

Thermally generated heterocyclic compounds such as furan have a higher sensory intensity in foam than pellets. Hexanal and 2-pentylfuran were described as having grassy and beany odors (Table 2). Several investigators found similar characteristics in soy protein isolates, textured soy proteins and soybean oil (3, 4, 9). Another lipid oxidation product, nonanal, was perceived as "oily, sweet and grassy" in pellets and as "oily and oxidized oil" in the resulting foam. Nonanal has been described as fatty, tallow-like, or floral attributes (9, 14).

1-Octen-3-ol was described as grassy or mushroom-like in an agreement with Aaslyng et al. (15). 1-Octen-3-ol has been reported in roasted chicory, oxidized soybean oil, prawn and sand-lobster. It is a lipid oxidation product (9). Enzyme-catalyzed oxidation in soybeans also might produce the compound (6).

The panelists detected a roasted and burned aroma associated 2,6-dimethylpyrazine in the foam (Table 2). Pyrazines are important volatiles in coffee, cooked meat and popcorn. The odors of pyrazines can range from roasted meat-like to nutty notes (9).

In order to reduce the undesirable volatile compounds from the soy protein-based foam, lipoxygenase-free soy protein isolates (i.e. as starting material) might be used to replace normal soy protein isolates. Lipoxygenases consist of three isoenzymes, L-1, L-2 and L-3, are found in soybeans and have been associated with the productions of beany and oxidized flavor in soy products (9, 16). The enzymatic oxidation and cleavage of polyunsaturated fatty acids in soybeans produce aldehydes and alcohols. Lipoxygenase-free soybean varieties have been shown to produce limited amounts of these off-odor compounds. In addition, temperature adjustment during extrusion can be used to minimize the products of thermal degradation. Odor masking agents also might be used to reduce the undesirable odor of soy protein-based plastics.

#### Acknowledgements

The authors acknowledged the funding provided in part by the Iowa Soybean Promotion Board and United Soybean Board; P. Mungara provided soy protein-based pellets and soy protein-based foam; Lie Lu provided technical assistance.

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No. <sup>a</sup>	Compound	Soy protein-based	Soy protein-based
	Taluana	penets	10ain
1		I	-1
2	Hexanal	Ŧ	+
3	Pentanol	—	+
4	Ethylbenzene	+	+
5	Styrene	+	+
6	Hexanol	+	+
7	2, 6-Dimethylpyrazine	_	+
8	2-Heptanone	+	+
9	Benzaldehyde	+	+
10	1-Octen-3-ol	+	+
11	2-Pentylfuran	+	+
12	Limonene	+	+
13	2-Ethylhexanol	+	+
14	3, 5-Octadien-2-one	+	+
15	3-Ethyl-2, 5-dimethylpyrazine	_	+
16	Nonanal	+	+
17	1, 2, 3, 4-Tetrahydronaphthalene	+	+
18	Naphthalene	+	+
19	2-Nonenal, (E)	+	+
20	2-Pentylthiophene	+	+
21	2-Decanone	+	+
22	Decanal	+	+

Table 1. Low Molecular-Weight Volatile Compounds Found in Soy Protein-Based Pellets and Soy Protein-Based Foams

<sup>a</sup>Numbers correspond to those in figures 1 and 2.

 $^{b}$  + detectable

	Average relative intensity <sup>a</sup>		ative intensity <sup>a</sup>	
Compound	Retention	Soy protein-	Soy protein-	Odor description
	time (min)	based pellets	based foam	
Toluene	1.2	2.6	2.4	Chemical solvent <sup>bc</sup> , burned plastic <sup>bc</sup> ,
				sour <sup>b</sup>
Hexanal	2.6	2.2	3.6	Grassy <sup>be</sup> , beany <sup>be</sup>
Hexanol	4.9	0.9	2.1	Earthy <sup>e</sup> , oily <sup>e</sup>
2,6-Dimethyl-	5.8		1.9	Roasted <sup>°</sup> , burned <sup>°</sup>
pyrazine				
1-Octen-3-ol	10.6	0.6	1.9	Grassy <sup>°</sup> , mushroom <sup>°</sup>
Benzaldehyde	11.5	0.8	0.9	
2-Pentylfuran	11.6	1.3	2.6	Grassy <sup>e</sup> , beany <sup>e</sup>
Limonene	14.4	1.2	2.1	Fruity <sup>c</sup> , fragrant <sup>c</sup> , sweet <sup>c</sup>
Nonanal	21.4	2.0	2.1	Oily <sup>be</sup> , oxidized oil <sup>e</sup> , grassy <sup>b</sup> , sweet <sup>b</sup>
Decanal	33.4	1.1	2.1	Oily <sup>c</sup> , green <sup>c</sup>

Table 2. Odors Identified from Soy Protein-Based Pellets and Soy Protein-Based Foams by Gas Chromatography-

#### Olfactometry

 $a_0 =$  undetectable; 1 = able to detect odor but unable to describe the odor; 2 = mild but able to describe the odor; 3 =

medium; 4 = moderately strong; 5 = strong.

<sup>b</sup>Soy protein-based pellets.

<sup>c</sup>Soy protein-based foam.



Figure 1. Total ion chromatogram of soy protein-based pellets



Figure 2. Total ion chromatogram of soy protein-based foams

## THE EFFECTS OF STORAGE TEMPERATURE AND FOOD pH'S ON THE FUNCTIONAL PROPERTIES OF FULL-FAT SOY FLAKES

A paper to be submitted to the Journal of the American Oil Chemists' Society Ee-Fah Chong and Lester A. Wilson

#### Abstract

Effects of storage temperature and food pH's on the foaming properties, oil absorption capacity and water-holding capacity of full-fat soy flakes were studied. Maximum foam expansion of soy flakes occurred at pH 5.5. Soy flakes stored at temperature above 25°C showed less stable foams. Soy flakes exhibited maximum oil absorption at 50°C in 7 days and showed maximum water-holding capacity at pH 9.0. Commercial full-fat soy flakes were generally most functional at pH values above their isoelectric point. No significant change was observed for foam expansion, oil absorption and water-holding capacity of soy flakes stored at 4°C for 28 days. High storage temperatures, i.e. 45 and 50°C, have more pronounced effects on the quality of soy flakes. Flake quality will be negatively affected if exposed to high temperatures during shipping, handling, and storage conditions.

#### Introduction

Soybeans and their products are widely used as functional and nutritional ingredients in food products. The quality of soybeans and soy flakes can be altered during shipment when the beans and flakes are exposed to adverse environmental conditions especially at elevated temperatures. The changes of functional properties in soybeans and soy flakes are critical in providing quality end products for food systems.

Functional properties of soy proteins, such as foaming properties, oil absorption capacity and water-holding capacity, are important factors in delivering desirable texture, flavor and stable characteristics to foods in order to be accepted by consumers. The properties can be affected by food formulation, food processing, packaging, storage and shipping conditions. These properties reflected the composition and conformational change of protein molecules and the interactions between proteins/fats/and carbohydrates that are affected by the temperature, pH, and protein concentration (1, 2).

Many studies have focused on the functional properties of soybean and soy protein products (3, 4, 5). Martin and Davis (4) found that doughnuts that contain high nitrogen solubility index (NSI) defatted soy flours absorbed less oil than that of the low NSI. Fleming et al. (6) found that water-holding of soy isolates were higher than that of soy flour and soy concentrates. Lin et al. (7) demonstrated that fat absorption increased as protein content increased. However, the effects of storage temperature and end use pH's on the functional properties of full-fat soy flakes are not widely studied.

The objectives of this study were to examine the effects of storage temperature and typical food/beverage pH level on foaming properties, oil absorption and water-holding capacity of full-fat soy flakes. This information will be important in controlling the quality of soy flakes during shipping and both short and long term storage.

#### **Materials and Methods**

#### Materials

Commercial full-fat soy flakes (Microsoy Flakes) were obtained from the Mycal Corporation of America (Jefferson, IA); reagents were of analytical grade and purchased from Fisher Scientific (Pittsburgh, PA).

#### Storage conditions

Bags of flakes were stored in a 4°C walk-in refrigerator until analysis. Full-fat soy flakes in 20-kg heat sealed polyethylene-paper lined bags were randomly numbered and stored in temperature-controlled incubators at 4, 25, 45 and 50°C. Flakes from whole bags were drawn at 7, 14, and 28 days of storage periods except soy flakes stored at 4°C which were drawn at day 0 and at 28 days. The samples were analyzed for foam expansion, foam stability, oil absorption capacity and water-holding capacity. After the completion of the first experiment, a second replication was initiated.

#### Foaming properties

Foam expansion and foam stability determinations were modified according to the method of Chen and Morr (1). Full-fat soy flakes were dispersed in distilled water to produce a 1% protein solution (w/v). Each solution was adjusted to pH 3.5, 5.5, 6.5, 7.0 or 9.0 with 0.1N HCl or NaOH. The aliquots were held at  $23\pm1^{\circ}$ C with constant agitation in a shaker for 1 hr. Foaming properties were measured by using Kitchen Aid model K5-SS mixer (St. Joseph, MI) equipped with stainless steel bowl and wire whip beater. Each solution was whipped at a speed setting of 8 for 5 min. The foam and the remaining solution

were transferred to a graduated cylinder. The foam volume was measured after whipping the solution at 5 min. This was considered the initial volume. Then, the foam and the remaining solution were held for an additional 55 min. The foam volume at 60 min was recorded, and the percent foam expansion and foam stability were calculated as follows:

Percent of foam expansion = (Volume foam at 5 min) 100/ Original solution volume Percent of foam stability = (Volume foam at 60 min/ Volume foam at 5 min) x 100

#### **Oil absorption capacity**

Oil absorption capacity was determined by the method of Onuma Okezie and Bello (8). One gram of each sample was homogenized at high speed in a blender (Waring-Whirl model 33BL79, New Hartford, CT) for 1 min with 50 mL corn oil (Mazola 100% Pure Corn Oil, Bestfoods, NJ). After being held at room temperature for 30 min, the samples were centrifuged at 3,000 rpm for 30 min. Density of oil was 0.92 g/mL. The oil absorption capacity was calculated and expressed as the grams of oil absorbed per gram of sample (as is basis):

Oil absorption capacity = (Total volume of oil – volume of oil free from absorption) x Oil density

#### Kinetic reactions of oil absorption capacity

The reaction kinetics was modeled using apparent first order kinetics. The rate constants of the reaction were determined in a semi-log function of time for each storage temperature. The activation energy was calculated by using the Arrhenius equation (9, 10).

The first order reaction can be expressed as (9):

$$-dc/dt = kc$$

where -dc/dt = rate of change of absorption capacity, c =existing absorption capacity, and k = first-order rate constant.

#### Water-holding capacity

Water-holding capacity was modified by the method of Hutton and Campbell (11). Five grams of soy flakes was transferred into 50-mL centrifuged tubes, and 40-mL of distilled water was added to each sample. Each solution was adjusted to pH 3.5, 5.5, 6.5, 7.0 or 9.0 with 0.1N HCl or NaOH. The mixtures were stirred for 30 sec then hold for 10 min. The aforementioned process was repeated seven times. After centrifuging the samples at 3,000 rpm for 25 min, the supernatants were decanted. The tubes were allowed to dry in an air oven for 25 min at 50°C. Then, the samples were cooled in the desiccator and weighed. The water-holding capacity was calculated as the difference between hydrated weight and original weight, and expressed as a percentage of the original dry weight of the sample.

#### Nitrogen solubility index

Nitrogen solubility index of full-fat soy flakes stored at 4°C, drawn initially as a control, was modified by the method of Qi et al. (3). For each 1% protein sample, the suspension was adjusted to pH 3.5, 4.5, 5.0, 5.5, 6.5, 7.0 or 9.0 with 0.1N HCl or NaOH. The suspensions were shaken for 2 hours at room temperature. Then, the samples were centrifuged at 9,500 rpm for 30 min. Nitrogen content in the supernatant was determined by Kjeldahl method (12).

#### Statistical analysis

The data were analyzed by using an analysis of variance for a split plot experiment in a randomized block design (13). Experimental results were analyzed by PROC MIXED with the Statistical Analysis System (14). Significance was defined at  $p \le 0.05$ .

#### **Results and Discussion**

#### Foaming properties of soy flakes

The foam expansion of the full-fat soy flakes stored at 4°C at day 0 is presented in Figure 1. Maximum foam expansion of soy flakes occurred at pH 5.5 with a minimum occurring at pH 3.5. This is in contrast to the foam expansion curve of alcohol-washed soy isolates which showed minimum foams in the isoelectric region, pH 4.5, and maximum at pH 2 and 9 (15). On the other hand, Garcia et al. (5) reported that foamability of soy protein is higher near the isoelectric point (pI) of the proteins. Protein molecules have minimum net charges near the isoelectric point. According to Damodaran (16), the amount of proteins adsorbed to the air-water interface increased at the pI. The interaction is due to the lack of repulsion between the interface and adsorbing molecules. The results indicated that the foaming activity of proteins is related to the physiochemical properties of the protein materials and the environmental conditions such as the type of processing (e.g. alcohol extraction) and method of preparation. Soy flakes contain more lipids, lecithins and carbohydrates than soy isolates. These compounds could alter the foaming properties of soy flakes which could account for the pH shifts compared to Eldridge et al. (15).

Foam expansion of soy flakes at pH 6.5 stored at 4°C, 25°C, 45°C and 50°C at day 28 were 22.8%, 27.5%, 30.2% and 32.9%, accordingly (Figure 2). The results demonstrated

that foam expansion of soy flakes at pH 6.5 increased with an increase in storage temperature. This might be due to partial unfolding of protein molecules exposing active sites that can interact at the air-water interface (16). In addition, oxidation of the protein and the lipid fraction would favor hydrophilic interactions. No differences in foam expansion were observed for full-fat soy flakes stored at 4°C for 28 days at various pH's. Soy flakes stored below 25°C could provide better quality. Food products in which soy proteins are used for foaming properties included whipped toppings and icing. However, foaming is not desirable in producing soymilk.

The influence of pH on foam stability of soy flakes at day 0 is shown in Figure 3. Full-fat soy flakes showed optimum foam stability at pH 5.5, which was near the isoelectric point of the flakes (Figure 4). The lack of repulsive interactions favors protein-protein interactions and formation of viscous film at the interface. Thus, the reaction provides stable foam (2, 17).

Soy flakes had zero foam stability at pH 3.5 and pH 9.0 (Figure 3). This is contrast to the behavior of alcohol-washed soybean proteins, which exhibited stable foams below pH 3.0 and above pH 6.5 (15). The results indicated that acidic or alkaline condition that maximize the net charge on protein molecules tends to reduce foam stability. Strong intramolecular electrostatic repulsion at extreme pH values decreases viscous film formation at the interface (16). Subsequent reduction in film formation disrupts the integrity of foams.

The foam stability curves showed that soy flakes stored at temperatures above 25°C were not capable of generating stable foams at day 14 (Figure 5). The stability of the foams decreased at high storage temperatures due to the increase in gas diffusion and drainage. The effect seen in the range of temperature studied probably could alter the folding of protein

molecules. At higher temperatures, noncovalent interactions are disrupted and the reaction might decrease the viscosity of the film (16). Thus, the results demonstrated that protein molecules were capable of developing stable foams at lower temperatures. Foaming properties of the soy flakes were affected significantly both by storage temperature and pH (p<0.05), as well as the pH-storage temperature interaction effect.

The kinetic reactions of foam expansion and foam stability were not fitted by zero, first or second-order reactions. The changes occurring during foaming were more complex. In addition to storage temperature, pH affected reactions in foaming. There were also possible interaction between pH and temperature in the simple system.

#### Oil absorption capacity of soy flakes

Oil absorption, expressed as g oil/g sample on the as-is basis, is shown in Figure 6. Oil absorption of the stored flakes at 4°C for 28 days was significantly lower (P<0.05) than that of 45°C and 50°C. However, there was no significant difference between 4°C and 25°C for the oil absorption of soy flakes. The oil absorption capacity of the flakes increased significantly as the storage temperature increased. Oil absorption capacity of soy flakes stored at 4°C did not change significantly over 28 days. The curves for the storage temperature of 45°C and 50°C were similar to each other.

At 28 days of storage, the maximum oil absorption of the flakes was at 50°C, followed by those at 45°C, 25°C and 4°C. The oil absorption slightly increased by heating. The results agreed with Lopez de Ogara et al (18). The rise of oil adsorption of soy flakes may be due to some denaturation of proteins especially samples stored at 50°C. It could promote the binding of oil by nonpolar side chains of protein molecules. The ability of soy flakes to retain oil may be used to incorporate in bakeries and comminuted meat systems. The property may improve the texture and mouth feel and decrease yield loss in foods.

#### Kinetic reactions of oil absorption capacity

The kinetics of oil absorption was best fitted to a first-order reaction for the pertinent temperature range. The apparent first-order rate constants (k) of the reaction at 25°C, 45°C and 50°C across 28 days were 4.53, 5.30 and 5.68 x 10<sup>-3</sup> day<sup>-1</sup>, respectively. The small differences between the reaction rate at each temperature and their low k values would indicate only minor changes during 28 days of storage. However, at the high temperatures (i.e., 45°C and 50°C) the reaction was completed by day 7. The reaction at 4°C and 25°C had still not reach the same level of fat absorption in 28 days as had the high temperatures in 7 days (Figure 6). Insufficient data was available to accurately calculated the k values for 45°C and 50°C after 7 days of storage, however, an estimated k value would be over five times higher than that calculated after 28 days of storage.

The activation energy of oil absorption in full-fat soy flakes measures the temperature sensitivity of the reaction (Arrhenius plot). The slope of straight line between the natural logarithm of rate constant versus 1/absolute temperature determined the activation energy ( $E_a$ ) (Figure 7). The estimated  $E_a$  of the reaction was 6.96 kJ/mole. Usually, activation energies of food reactions are approximately 42 to 209 kJ/mol (19). The low activation energy indicated low to no temperature-dependence of the oil absorption in the flakes at the temperature range of 25°C to 50°C during the 28 days of storage although this activation

energy is lower than it should be due to the high reaction rate at 45°C and 50°C at day 7. The activation energy should be recalculated after a more accurate reaction rate at high temperatures can be determined.

#### Water-holding capacity of soy flakes

The effects of pH on the water-holding capacity of full-fat soy flakes at day 0 are shown in Figure 8. Water-holding capacity was lowest at pH 6.5 and highest at pH 9.0. The high end of the pH range tends to disrupt the quaternary structure of soy proteins, exposing the hydrophilic sites of protein molecules allowing them to interact with water molecules (16).

After 28 days of storage all temperature treatments, with the exception of 4°C, still exhibited minimum water holding capacity at pH 6.5 (Figure 9). At 4°C, the minimum has shifted from pH 6.5 to pH 7.0.

The independent effect of pH on the water-holding capacity was significant at the level of P < 0.05, as was the pH-storage temperature interaction. However, no significant difference was found in the storage temperatures of soy flakes. The water-holding capacity of full-fat soy flakes showed greater responsive to pH than the storage temperatures. The kinetic reaction behavior for water-holding capacity was similar to the aforementioned condition in foaming. Water-holding of the proteins can be desirable in many food applications. For example, the property is important in moisture retention in meats or bakeries in order to enhance mouth feel and flavor in the foods.

In the simple system, the variations of pH and storage temperature have imposed effects on the foaming properties, oil absorption and water-holding capacity of the full-fat soy flakes. This effect was more prominent at higher temperatures and at pH 3.5 and pH 9.0 of soy flakes. High temperature and relative humidity storage conditions of soybeans, and full-fat soy flakes caused significant decreases in the quality of soymilk and tofu (20, 21). The quality changes included textural and appearance characteristics of the soymilk and tofu. The results suggested that storage temperatures below 25°C could well maintain the functional properties of properly packaged commercial soy flakes. The transit times during shipment of up to four weeks at low temperatures could provide optimum quality in soy flakes. Foaming capacity, oil absorption and water-holding capacity are some of the crucial properties used in the processing of sausages, beverages, cakes, simulated meat, and soy foods. Further research of the functional properties that related to the actual food product is needed to increase the use of soy flakes.

#### Acknowledgements

We acknowledged the funding provided by the Mycal Corporation of America; P. Dixon provided statistics assistance.

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Figure 1. The influence of pH on the foam expansion of full-fat soy flakes at  $4^{\circ}C$  at day 0.



Figure 2. The influence of pH on the foam expansion of full-fat soy flakes at day 28 at various storage temperatures:  $4^{\circ}C(\bullet)$ ;  $25^{\circ}C(\bullet)$ ;  $45^{\circ}C(\Delta)$ ;  $50^{\circ}C(\Box)$ .



Figure 3. The influence of pH on the foam stability of full-fat soy flakes at  $4^{\circ}C$  at day 0.



Figure 4. Nitrogen solubility index of full-fat soy flakes



Figure 5. The influence of pH on the foam stability of full-fat soy flakes at day 14 at various storage temperatures:  $25^{\circ}C(\blacklozenge)$ ;  $45^{\circ}C(\Delta)$ ;  $50^{\circ}C(\Box)$ .



Figure 6. Oil absorption capacity of full-fat soy flakes at various storage temperatures:  $4^{\circ}C(\bullet)$ ;  $25^{\circ}C(\bullet)$ ;  $45^{\circ}C(\Delta)$ ;  $50^{\circ}C(\Box)$ .



Figure 7. Arrhenius plot for oil absorption capacity of full-fat soy flakes



Figure 8. The influence of pH on the water-holding capacity of full-fat soy flakes at  $4^{\circ}$ C at day 0.



Figure 9. The influence of pH on the water-holding capacity of full-fat soy flakes at day 28 at various storage temperatures:  $4^{\circ}C(\bullet)$ ;  $25^{\circ}C(\bullet)$ ;  $45^{\circ}C(\Delta)$ ;  $50^{\circ}C(\Box)$ .

#### **GENERAL CONCLUSIONS**

Commercial soy protein isolates were thermally extruded at 100°C to produce pellets. The pellets were extruded at 150-160°C to make soy protein-based foam. The volatiles were stripped off and concentrated on a Tenax adsorbent, followed by gas chromatography-mass spectrometry (GC-MS) to identify the volatiles from the pellets and foams. The volatiles were mainly aldehydes, alcohols, ketones, aromatic hydrocarbons and pyrazines. Gas chromatography-olfactometry revealed that the strongest odors could be attributed to toluene and hexanal. The intensities of the odorous compounds increased with extrusion temperature. Pyrazines, 2,6-dimethylpyrazine and 3-ethyl-2, 5-dimethylpyrazine, were found only in the soy protein-based foam.

In order to reduce the undesirable compounds, lipoxygenase-free soy protein isolates could be used to replace the normal soy proteins used for the production of pellets and foams. This would reduce some of the bound or entrapped aldehydes, alcohols, and ketones produced by the lipoxygenase enzyme. In addition, masking agents can be used to reduce undesirable odor of the soy-based plastics.

Foaming properties, oil absorption and water-holding capacities are part of the essential functional properties in delivering texture, flavor, formation and stabilization of emulsions in food systems. Functional properties are not only critical in determining the quality of the end products, but also in facilitating the manufacturing of the products. Fullfat soy flakes can be used to produce soy flours, grits and soy foods such as soymilk and tofu. They may also be used in products intended for bakeries or animal feed.

The pH, storage temperature and pH-storage temperature interactions influenced the functional properties of soy flakes. Higher storage temperature increased the oil absorption

of the flakes. These would be undesirable in deep fat fried food such as doughnuts. Higher temperature storage also increased foam expansion which could be detrimental to soy food production (e.g., soymilk). The results indicated that full-fat soy flakes should be stored at temperatures below 25°C in order to maximize storage stability in the flakes and their use in foods and beverages. The pH of the foods or beverages is important because it sets the protein solubility and water-holding capacity of the added flakes. This in turn influences other functional and textural properties of the foods (e.g., high protein beverages, tofu, meat products).

Storage and transit times during shipment of up to four weeks at low temperatures could provide or maintain optimum quality in soy flakes. These interactions can provide implications for foods such as soymilk, tofu, bakery products or simulated meat that contain full-fat soy flakes or soy products derived from the flakes. This information could be used to develop specifications and recommendations for the optimum storage and transit conditions of full-fat soy flakes and potentially other similar high protein full-fat products.

#### ACKNOWLEDGEMENTS

I would like to express my heartfelt gratitude and appreciation to my major professors, Dr. Jay-Lin Jane and Dr. Lester A. Wilson, for their guidance, support, encouragement and patience throughout my graduate work at Iowa State University.

I am also grateful to my committee member, Dr. Philip Dixon for his assistance in data analysis and Dr. Earl G. Hammond for his unreserved help in GC-MS analysis.

Appreciation is extended to Sophia Lee, Julia Jiang, Safir Moizuddin, graduate students and undergraduate students in Dr. Jay-Lin Jane, Dr. Earl G. Hammond and Dr. Lester A. Wilson research groups for their help and friendship throughout my study.

Finally, I would like to thank my family for their loving support and encouragement during my studies at Iowa State University.